

**Data Evaluation Report on the terrestrial field dissipation of pinoxaden**

PMRA Submission Number {.....}

EPA MRID Number 46203025

**Data Requirement:**

PMRA Data Code:  
EPA DP Barcode: D300622  
OECD Data Point:  
EPA Guideline: 164-1

**Test material:** NOA-407855**End Use Product name:** NOA-407855 120EC**Concentration of a.i.:** 12%**Formulation type:** Emulsifiable concentrate**Active ingredient****Common name:** Pinoxaden.**Chemical name**

**IUPAC:** 8-(2,6-Diethyl-p-tolyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]-oxadiazepin-9-yl 2,2-dimethylpropionate.

**CAS name:** 8-(2,6-Diethyl-4-methylphenyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl 2,2-dimethylpropanoate.  
8-(2,6-Diethyl-4-methylphenyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl ester 2,2-dimethylpropanoic acid.

**CAS No:** 243973-20-8.

**Synonyms:** NOA-407855.

**SMILES string:** O=C1C(=C(N2N1CCOCC2)OC(=O)C(C)(C)C)c1c(cc(cc1CC)C)CC.

**Primary Reviewer:** Dan Hunt

Dynamac Corporation

**Signature:****Date:****QC Reviewer:** Joan Harlin

Dynamac Corporation

**Signature:****Date:****Secondary Reviewer:** Ibrahim Abdel-Saheb

Environmental Risk Branch II

Environmental Fate and Effects Division (7507C)

**Signature:****Date:****Company Code:****Active Code:****Use Site Category:****EPA PC Code:** 147500

**CITATION:** Speth, R.M. 2004. Terrestrial field dissipation of NOA-407855 120EC on bare soil and winter wheat in Texas. Unpublished study performed by Syngenta Crop Protection Inc., Greensboro, NC, Waterborne Environmental, Inc., Leesburg, VA (field management), Great Plains Crop Sciences, Groom, TX (field research facility), Agvise Laboratories, Northwood, ND



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**Test material:** NOA-407855

**End Use Product name:** NOA-407855 120EC

**Concentration of a.i.:** 12%

**Formulation type:** Emulsifiable concentrate

## Active ingredient

Common name: Pinoxaden.

Chemical name

IUPAC: 8-(2,6-Diethyl-p-tolyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]-oxadiazepin-9-yl 2,2-dimethylpropionate.

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**Primary Reviewer:** Dan Hunt  
Dynamac Corporation

**Signature:** Dan Hunt  
**Date:** 11/30/04

**QC Reviewer:** Joan Harlin  
Dynamac Corporation

**Signature:** Joan Harlin  
**Date:** 11/30/04

**Secondary Reviewer:** Ibrahim Abdel-Saheb  
EPA

**Signature:**  
**Date:**

**Company Code:**

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(soil characterization), and Central California Research Laboratories, Inc., Fresno, CA (analytical laboratory), and sponsored and submitted by Syngenta Crop Protection, Inc., Greensboro, NC.

Syngenta Number: 1297-01. Study initiation October 12, 2001 and completion January 22, 2004.

Final report issued January 22, 2004.

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### EXECUTIVE SUMMARY:

Soil dissipation/accumulation of pinoxaden (8-(2,6-diethyl-*p*-tolyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-*d*][1,4,5]oxadiazepin-9-yl 2,2-dimethylpropionate; NOA-407855) under US field conditions was conducted in a bare ground plot and a plot cropped with winter wheat at one site in Armstrong County, Texas (Ecoregion not reported). The experiment was carried out in accordance with the USEPA Pesticide Assessment Guidelines Subdivision N, §164-1 and in compliance with the USEPA FIFRA (40 CFR, Part 160) GLP standard. Pinoxaden was broadcast once at a target rate of 0.077 kg a.i./ha (0.0687 lb a.i./A) onto two plots (one bare ground and one cropped with winter wheat) of sandy loam soil (0-15 cm) that were each divided into three replicate plots measuring approximately 6 x 27 m. Pinoxaden was applied with the safener, cloquintocet-mexyl (CGA-185072). The winter wheat was at the 4-leaf growth stage at the time of test substance application. The target application rate corresponds to 110% of the current maximum annual rate for control of grass weeds in small grain crops. Rainfall was supplemented with irrigation to reach 199% of the 30-year average rainfall. The two treated plots were located approximately 0.9 m apart from each other and the nearest control plot was located approximately 25 m away from the treated plots.

Application monitors (petri dish tops containing filter paper) were placed in both the bare ground and cropped plots to verify the application rate; however, the application monitors samples were not analyzed. Field spikes were prepared in triplicate for pinoxaden and its transformation products NOA-407854 and NOA-447204 by fortifying 0-15 cm depth control soil samples with each analyte at 10 ppb on October 23, 2002; however, the field spike samples were not analyzed.

Soil samples were collected from the treated bare ground and cropped plots at -3, 0, 0.5, 1, 3, 7, 14, 21, 31, 38, 60, 91, 124, 185, 274, 389, 458, and 546 days following the test application to a depth of 0-120 cm (except day-0 and day-0.5 samples which were collected to a depth of 15 cm). Soil samples were analyzed for pinoxaden and its transformation products NOA-407854 (7H-pyrazolo[1,2-*d*][1,4,5]oxadiazepine-7,9(8H)-dione, 8-(2,6-diethyl-4-methylphenyl)tetrahydro-) and NOA-447204 (7H-pyrazolo[1,2-*d*][1,4,5]oxadiazepine-7,9(8H)-dione, 8-(2,6-diethyl-4-methylphenyl)tetrahydro-8-hydroxy-) and the safener, cloquintocet-mexyl and its transformation product CGA-153433. Soil samples (20 g) were extracted by sonicating with 60 mL of acetone:water (60:40, v:v) for 15 minutes and shaking for 45 minutes. An aliquot of the extract was passed through a 2-gram aminopropyl SPE column and a Nexus Absolut SPE column and analyzed for each analyte by LC/MS/MS. The LOQ for all analytes in soil was 0.5 ppb. Soil samples were stored frozen for up to 347 days (bare ground plot) and 351 days (cropped plot) prior to analysis.

In the bare ground plot, the measured zero-time concentration in the 0-15 cm soil depth was 5.9 ppb, which is 15% of the applied rate (reviewer-calculated based on a theoretical day-0 concentration of 0.039 mg a.i./kg in the 0-15 cm soil depth). The day-0 concentration of pinoxaden + NOA-407854 (after converting to parent equivalents) was 61% of the applied rate (reviewer-calculated). Following day 0, pinoxaden decreased to 2.5 ppb by 1 day, and was last detected above the LOQ at 0.61 ppb at 21 days posttreatment. Pinoxaden was not detected above

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the LOQ in soil below the 0-15 cm depth. The major transformation product **NOA-407854** was detected in the 0-15 cm soil depth at a maximum concentration of 14 ppb from 0 to 1 day (which is 45.4% of the applied pinoxaden, after converting to parent equivalents), then decreased to 8.5 ppb by 7 days, and was last detected above the LOQ at 1.3 ppb at 31 days posttreatment. NOA-407854 was not detected above the LOQ in soil below the 0-15 cm depth. The registrant-calculated half-life value for NOA-407854 was 10 days, calculated using non-linear regression. The minor transformation product **NOA-447204** was detected in the 0-15 cm soil depth at a maximum concentration of 2.3 ppb at 21 days (which is 7.2% of the applied pinoxaden, after converting to parent equivalents), and was last detected at 0.62 ppb at 124 days posttreatment. NOA-447204 was detected in the 15-30 cm and 30-45 cm soil depths at 0.75-1.4 ppb (31-124 days) and 0.52-0.58 ppb (31, 91, and 124 days), respectively.

In the cropped plot, the maximum measured concentration in the 0-15 cm soil depth was 7.6 ppb at day 0, which is 19% of the applied rate (reviewer-calculated based on a theoretical day-0 concentration of 0.040 mg a.i./kg in the 0-15 cm soil depth). The maximum concentration of pinoxaden + NOA-407854 (after converting to parent equivalents) was 59% of the applied rate at 0.5 days posttreatment (reviewer-calculated). Following day 0, pinoxaden decreased to 3.7 ppb by 1 day and was last detected above the LOQ at 0.80 ppb at 21 days posttreatment. Pinoxaden was not detected above the LOQ in soil below the 0-15 cm depth. The major transformation product **NOA-407854** was detected in the 0-15 cm soil depth at a maximum concentration of 13 ppb at 0.5 and 3 days (which is 41.1% of the applied pinoxaden, after converting to parent equivalents), then decreased to 6.6 ppb by 7 days, and was last detected above the LOQ at 1.7 ppb at 21 days posttreatment. NOA-407854 was not detected above the LOQ in soil below the 0-15 cm depth. The registrant-calculated half-life value for NOA-407854 was 8.1 days, calculated using non-linear regression. The minor transformation product **NOA-447204** was detected in the 0-15 cm soil depth at a maximum concentration of 1.7 ppb at 3 and 7 days (which is 5.1% of the applied pinoxaden, after converting to parent equivalents), and was last detected at 0.50 ppb at 185 days posttreatment. NOA-447204 was detected in the 15-30 cm and 30-45 cm soil depths at 0.50-1.1 ppb (31-185 days) and 0.51-0.79 ppb (31 and 38 days), respectively.

Under field conditions at the test site in the bare ground plot, the reviewer-calculated half-life of pinoxaden in soil under terrestrial field conditions was 7 days ( $r^2 = 0.73$ ), calculated using linear regression analysis performed on a plot of ln-transformed concentrations vs. time and the equation  $t_{1/2} = \ln 2 / k$ , where  $k$  is the rate constant (the observed half-life was approximately 1 day). A DT90 value was not calculated. The total carryover of residues of pinoxaden and its transformation products was 0% of the applied at 389 days posttreatment (the last sampling interval at which samples were analyzed), based on the target application rate.

Under field conditions at the test site in the cropped plot, the reviewer-calculated half-life of pinoxaden in soil under terrestrial field conditions was 6 days ( $r^2 = 0.80$ ), calculated using linear regression analysis performed on a plot of ln-transformed concentrations vs. time and the equation  $t_{1/2} = \ln 2 / k$ , where  $k$  is the rate constant (the observed half-life was approximately 1 day). A DT90 value was not calculated. The total carryover of residues of pinoxaden and its transformation products was 0% of the applied at 389 days posttreatment (the last sampling

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interval at which samples were analyzed), based on the target application rate.

The major route of dissipation of pinoxaden under terrestrial field conditions was transformation.

### RESULTS SYNOPSIS

Location/soil type: Near Groom, Texas/sandy loam-loam (0-45 cm) over clay loam (45-105 cm).

Half-lives (reviewer-calculated):

Bare ground plot: 7 days ( $r^2 = 0.73$ ; the observed half-life was approximately 1 day).

Cropped plot: 6 days ( $r^2 = 0.80$ ; the observed half-life was approximately 1 day).

Major transformation products detected:

NOA-407854 (7H-pyrazolo[1,2-d][1,4,5]oxadiazepine-7,9(8H)-dione, 8-(2,6-diethyl-4-methylphenyl)tetrahydro-).

Dissipation routes: Transformation.

**Study Acceptability:** This study is classified **supplemental** and does not satisfy the guideline requirement for a terrestrial field dissipation study because the concentration of pinoxaden in the field samples at the time of analysis may not accurately represent the concentration in the field at the time of sampling, since it is possible that pinoxaden degraded in the samples during their prolonged frozen storage prior to analysis. No additional terrestrial field dissipation study is required.

### I. MATERIALS AND METHODS

**GUIDELINE FOLLOWED:** The study was conducted according to USEPA Pesticide Assessment Guidelines Subdivision N, 164-1. One significant deviation from EPA Subdivision N, 164-1 noted was:

The concentration of pinoxaden in the field samples at the time of analysis may not accurately represent the concentration in the field at the time of sampling, since it is possible that pinoxaden degraded in the samples during their prolonged frozen storage prior to analysis. This does not affect the validity of the study.

**COMPLIANCE:** The study was conducted in compliance with USEPA FIFRA (40 CFR Part 160) Good Laboratory Practice standards. Signed and dated GLP Compliance, Quality Assurance, and Data Confidentiality statements were provided.

#### A. MATERIALS:

**1. Test Material** Pinoxaden (NOA-407855).

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### Chemical Structure

of the active ingredient: See DER Attachment 1.

### Description:

Emulsifiable concentrate.

### Storage conditions of test chemicals:

$\leq 20^{\circ}\text{C}$  (Appendix 2, Figure 1).

### Physico-chemical properties of pinoxaden.

Parameter	Values	Comments
Water solubility	Not reported	
Vapour pressure/volatility	Not reported	
UV absorption	Not reported	
pKa	Not reported	
$K_{ow}/\log K_{ow}$	Not reported	
Stability of Compound at room temperature	Not reported	

**2. Test site:** The test site was located approximately four miles southwest of Groom, Texas (in Armstrong County), an area representative geographically and climatically of the Central Great Plains region of the United States where winter wheat is grown (Appendix 1, Figures 1-2). The treated and control plots had been fallow in 2000 and had been used to grow dry wheat in 1998 and 1999 (Appendix 1, Table 2). No chemicals had been applied during the five years prior to the application of the test substance.

Table 1: Geographic location, site description and climatic data at the study site.

Details		Test site
Geographic coordinates	Latitude	Not reported
	Longitude	Not reported
	Province/State	Texas
	Country	US
	Ecoregion	Not reported
Slope Gradient		0-2% (est.)
Depth to ground water (m)		>1.8 m

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Details	Test site
Distance from weather station used for climatic measurements	Daily precipitation, minimum and maximum air temperatures and soil temperature (at 2-inch depth) were recorded on-site and evapotranspiration data were recorded from a weather station located approximately 20 miles north of the test site.
Indicate whether the meteorological conditions before starting or during the study were within 30 year normal levels (Yes/No). If no, provide details.	Total water input (rainfall plus irrigation) during the study period (546 days) was 52.17 inches or 199% of the 30-year average rainfall.

Data were obtained from pp. 15, 17 and 22, and Appendix 1, Table 7, p. 82 of the study report.

Table 2: Site usage and management history for the previous three years.

Use	Year	Test site
Crops grown	Previous year	Fallow
	2 years previous	Wheat
	3 years previous	Wheat
Pesticides used	Previous year	None
	2 years previous	None
	3 years previous	None
Fertilizers used	Previous year	Not reported
	2 years previous	Not reported
	3 years previous	Not reported
Cultivation methods, if provided (eg., Tillage)	Previous year	Not reported
	2 years previous	Not reported
	3 years previous	Not reported

Data were obtained from Appendix 1, Table 2, p. 77 of the study report.



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### 3. Soils:

Table 3: Properties of the soil from the bare ground plot (Bippus fine sandy loam).

Property	Depth (cm)							
	0-15	15-30	30-45	45-60	60-75	75-90	90-105	105-120
Textural classification	SL	SL	L	CL	CL	CL	CL	CL
% sand	52	54	44	42	38	36	38	30
% silt	38	28	32	30	28	30	32	34
% clay	10	18	24	28	34	34	30	36
pH	8.2	8.0	8.0	8.0	8.0	8.1	8.1	8.1
Total organic matter (%)	1.0	1.3	1.1	1.0	1.0	0.9	0.8	0.8
CEC (meq/100 g)	13.8	17.5	22.3	24.4	27.6	26.3	23.3	25.6
Bulk density (g/cm <sup>3</sup> )	1.32	1.20	1.20	1.16	1.18	1.21	1.20	1.13
Moisture at 1/3 atm (%)	13.2	20.7	23.9	26.1	24.8	23.9	24.0	25.8
Taxonomic classification (e.g., ferro-humic podzol)	Fine-loamy, mixed, superactive, thermic Cumulic Haplustolls							
Soil mapping unit	Not available.							

Data were obtained from pp. 15-16 and Appendix 1, Table 3, p. 78 of the study report. SL = Sandy loam, L = Loam, CL = Clay loam. The taxonomic classification was obtained from the NRCS for the Bippus soil series.

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**Table 4: Properties of the soil from the cropped plot (Bippus fine sandy loam).**

Property	Depth (cm)							
	0-15	15-30	30-45	45-60	60-75	75-90	90-105	105-120
Textural classification	SL	L	L	CL	CL	CL	CL	SCL
% sand	68	46	42	40	40	40	40	46
% silt	22	34	32	30	28	26	30	26
% clay	10	20	26	30	32	34	30	28
pH	8.1	8.0	8.0	8.0	8.0	8.1	8.1	8.1
Total organic matter (%)	1.1	1.4	1.2	1.0	1.0	0.9	0.8	0.6
CEC (meq/100 g)	13.8	21.1	23.2	26.1	28.1	28.3	25.9	21.3
Bulk density (g/cm <sup>3</sup> )	1.29	1.20	1.18	1.17	1.19	1.21	1.23	1.24
Moisture at 1/3 atm (%)	13.5	21.9	31.0	30.9	28.7	27.8	27.2	25.4
Taxonomic classification (e.g., ferro-humic podzol)	Fine-loamy, mixed, superactive, thermic Cumulic Haplustolls							
Soil mapping unit	Not available.							

Data were obtained from pp. 15-16 and Appendix 1, Table 3, p. 78 of the study report. SL = Sandy loam, L = Loam, CL = Clay loam, SCL = Sandy clay loam The taxonomic classification was obtained from the NRCS for the Bippus soil series.

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## B. EXPERIMENTAL DESIGN:

### 1. Experimental design:

Table 5: Experimental design.

Details		Bare ground plot	Cropped plot
Duration of study		546 days	546 days
Uncropped (bare) or cropped		Bare	Cropped
Control used (Yes/No)		Yes	Yes
No. of replications	Controls	One	One
	Treatments	Three	Three
Plot size (L x W m)	Control	13.7 x 10.6 m	13.7 x 10.6 m
	Treatment	27.4 x 6.1 m/replicate plot	27.4 x 6.1 m/replicate plot
Distance between control plot and treated plot		24.6 m to the nearest treated plot	24.6 m to the nearest treated plot
Distance between treated plots		0.9 m between replicate plots	0.9 m between replicate plots
Application rate(s) used (g a.i./ha)		77 g a.i./ha	77 g a.i./ha
Was the maximum label rate per ha used in study? (Yes/No)		The application rate was 110% of the current maximum annual rate for small grain crops.	The application rate was 110% of the current maximum annual rate for small grain crops.
Number of applications		One	One
Application Date(s) (dd mm yyyy)		19/10/2001	19/10/2001
For multiple applications, application rate at Day 0 and at each application time (mg a.i./kg soil) <sup>1</sup>		0.039 mg a.i./kg soil	0.040 mg a.i./kg soil
Application method (eg., spraying, broadcast etc.)		Broadcast	Broadcast
Type of spray equipment, if used		Tractor-mounted boom sprayer with 16 XR8004 flat fan nozzles. The spray height was 20 inches above the soil.	Tractor-mounted boom sprayer with 16 XR8004 flat fan nozzles. The spray height was 20 inches above the soil.
Total volume of spray solution applied/plot OR total amount broadcasted/plot <sup>2</sup>		21,162 mL	21,162 mL
Identification and volume of carrier (e.g., water), if used		Water	Water

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Details		Bare ground plot	Cropped plot
Name and concentration of co-solvents, adjuvants and/or surfactants, if used		Merge™ (cloquintocet-mexyl; CGA-185072) at 0.70%.	Merge™ (cloquintocet-mexyl; CGA-185072) at 0.70%.
Indicate whether the following monthly reports were submitted:			
Precipitation		Yes	Yes
Average minimum and maximum air temperature		Yes	Yes
Average minimum and maximum soil temperature		Yes (2-inch depth)	Yes (2-inch depth)
Average annual frost-free periods		No	No
Indicate whether the Pan evaporation data were submitted		No, evapotranspiration data were reported.	No, evapotranspiration data were reported.
Meteorological conditions during application	Cloud cover	0%	0%
	Temperature (°C)	10.6°C	10.6 °C
	Relative humidity	50%	50%
	Wind speed	0-4 mph, NW	0-4 mph, NW
	Sunlight (hr)	Not reported	Not reported
Pesticides used during study:			
name of product/a.i concentration:		2,4-D + Glyphosate	2,4-D + Glyphosate
amount applied:		Three times at 3 pt/A	Three times at 3 pt/A
application method:		Not reported	Not reported
name of product/a.i concentration:		2,4-D	2,4-D
amount applied:		Once at 2 pt/A	Once at 2 pt/A
application method:		Not reported	Not reported
Supplemental irrigation used (Yes/No)		Yes	Yes
If yes, provide the following details:			
No. of irrigation:		8	8
Interval between irrigation:		20-131 days	20-131 days
Amount of water added each time:		1.14-2.70 inches	1.14-2.70 inches
Method of irrigation:		Overhead sprinkler	Overhead sprinkler
Indicate whether water received through rainfall + irrigation equals the 30 year average rainfall (Yes/No)		Yes	Yes
Were the application concentrations verified? (Briefly describe in Section 2, if used)		Yes	Yes
Were field spikes used? (Briefly describe in Section 3, if used)		Yes	Yes
Good agricultural practices followed (Yes or No)		Not reported	Not reported

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Details	Bare ground plot	Cropped plot
Indicate if any abnormal climatic events occurred during the study (eg., drought, heavy rainfall, flooding, storm etc.)	The plot received 5.40 inches of rainfall on October 7, 2002 and 10.05 inches of rainfall for the month of October 2002.	The plot received 5.40 inches of rainfall on October 7, 2002 and 10.05 inches of rainfall for the month of October 2002.
If cropped plots are used, provide the following details:  Plant - Common name/variety:  Details of planting:  Crop maintenance (eg., fertilizers used):	N/A	Winter wheat/ Weathermaster Planted on September 18, 2001 at a seeding rate of 70 lb/A. The wheat crop was harvested on June 27, 2002.
Volatilization included in the study (Yes/No) (if included, describe in Section 4)	No	No
Leaching included in the study (Yes/No) (if included, describe in Section 5)	Yes	Yes
Run off included in the study (Yes/No) (if included, describe in Section 6)	No	No

Data were obtained from pp. 15-18 and Appendix 1, Tables 6-10, pp. 81-85, Figures 3-5, pp. 93-95 and pp. 98-116 in the study report.

<sup>1</sup> The application rate at day 0 was calculated by the reviewer based on the target application rate of 77 g a.i./ha and using a site bulk density of 1.32 g/cm<sup>3</sup> for the bare ground plot and 1.29 g/cm<sup>3</sup> for the cropped plot, and a soil depth of 15 cm for both plots. The registrant calculated a theoretical value of 34 ppb for the bare ground and cropped plots, based on a bulk density of 1.50 g/cm<sup>3</sup> (p. 24).

<sup>2</sup> Reviewer-calculated based on the number of tractor passes (three passes of 90 ft = 270 ft total pass), the target tractor speed of 4.13 ft/sec and the sprayer output data (323.7 ml/sec; pp. 17-18 and Appendix 1, Table 8, p. 83).

**2. Application Verification:** Fifteen petri dish tops (15-cm diameter) containing filter paper (15-cm diameter) were randomly placed in the bare ground and cropped plots prior to application. After application, the petri dish tops were collected and shipped frozen to the analytical laboratory.

The concentration of pinoxaden in the spray tank solutions was verified by collecting six 10-mL spray solution samples from the tank mix (three samples before application and three after application).

**3. Field Spiking:** Field spikes were prepared in triplicate for pinoxaden and its transformation products NOA-407854 and NOA-447204 by fortifying 0-15 cm depth control soil samples with each analyte at 10 ppb on October 23, 2002 (369 days posttreatment); however, the field spike samples were not analyzed (Appendix 1).

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**4. Volatilization:** Volatilization was not measured.

**5. Leaching:** Fifteen cores were collected from the treated bare ground and cropped plots at -3, 0, 0.5, 1, 3, 7, 14, 21, 31, 38, 60, 91, 124, 185, 274, 389, 458, and 546 days following the test application to a depth of 120 cm (except day-0 and day-0.5 samples which were collected to a depth of 15 cm) to determine the mobility of the test substance in the soil profile. Five cores were collected from the two control plots at each sampling interval with the exception of day 0.5.

**6. Run off:** Run off was not studied.

**7. Supplementary Study:** A soil freezer storage stability study was conducted as a separate study and submitted as MRID 46203022. In the storage stability study, soil samples from the terrestrial field dissipation study site were fortified with pinoxaden and its transformation products NOA-407854 and NOA-447204 to achieve a concentration of 5 ppb for each analyte. Samples were analyzed following up to a maximum of 12 months of storage, with additional samples scheduled for analysis following up to 18 months.

### 8. Sampling:

Table 6: Soil sampling.

Details	Bare ground plot	Cropped plot
Method of sampling (random or systematic)	Random	Random
Sampling intervals	-3, 0, 0.5, 1, 3, 7, 14, 21, 31, 38, 60, 91, 124, 185, 274, 389, 458, and 546 days following application.	-3, 0, 0.5, 1, 3, 7, 14, 21, 31, 38, 60, 91, 124, 185, 274, 389, 458, and 546 days following application.
Method of soil collection (eg., cores)	Cores	Cores
Sampling depth	120 cm (48 inches) <sup>1</sup>	120 cm (48 inches) <sup>1</sup>
Number of cores collected per plot	15/treated plot (5 replicates from each of the 3 subplots) and 5/control plot	15/treated plot (5 replicates from each of the 3 subplots) and 5/control plot
Number of segments per core	Eight	Eight
Length of soil segments	15 cm	15 cm
Core diameter (Provide details if more than one width)	5.4 cm (2 1/8 inches) for the 0-15 cm depth samples and 3.0 cm (1.2 inches) for the 15-120 cm depth samples.	5.4 cm (2 1/8 inches) for the 0-15 cm depth samples and 3.0 cm (1.2 inches) for the 15-120 cm depth samples.

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Details	Bare ground plot	Cropped plot
Method of sample processing, if any	Soil samples were composited by subplot and depth to form three samples for each interval/depth combination and then homogenized with dry ice.	Soil samples were composited by subplot and depth to form three samples for each interval/depth combination and then homogenized with dry ice.
Storage conditions	Frozen	Frozen
Storage length	Up to 347 days.	Up to 351 days.

Data were obtained from pp. 19-21 and Appendix 1, Table 10, p. 85, and Appendix 2, p. 186; Table 7, pp. 212-220; and Table 14, pp. 251-261 of the study report.

<sup>1</sup> Excludes day-0 and day-0.5 samples which were collected to a depth of 15 cm (6 inches).

**9. Analytical Procedures:** The analytical method used for determining pinoxaden and its transformation products NOA-407854 (7H-pyrazolo[1,2-d][1,4,5]oxadiazepine-7,9(8H)-dione, 8-(2,6-diethyl-4-methylphenyl)tetrahydro-) and NOA-447204 (7H-pyrazolo[1,2-d][1,4,5]oxadiazepine-7,9(8H)-dione, 8-(2,6-diethyl-4-methylphenyl)tetrahydro-8-hydroxy-) and the safener, cloquintocet-mexyl (CGA-185072; acetic acid, [(5-chloro-8-quinolinyl)oxy]-, 1-methylhexyl ester) and its transformation product CGA-153433 (acetic acid, [(5-chloro-8-quinolinyl)oxy]-) in soil was Syngenta method No. 35-01 (Appendix 2, Figure 1). The LOQ for all analytes in soil was 0.50 ppb.

Soil samples (20 g) were extracted by sonicating with 60 mL of acetone:water (60:40, v:v) for 15 minutes and shaking for 45 minutes (Appendix 2; Figure 2). The samples were centrifuged and the extraction was repeated with 20 mL of extraction solution. The extract was transferred to a 2-gram aminopropyl SPE column. The eluate was collected and the acetone was removed by rotary evaporation. After the addition of a 5-mL aliquot of brine solution and 25 mL of water, the extract was transferred to a 1-gram Nexus Absolut SPE column. Analytes were eluted from the SPE cartridge using 2% formic acid in methanol, and the methanol was removed by rotary evaporation. The eluent was brought up to 20 mL with water and analyzed by HPLC (Waters Symmetry® Shield RP8 column, 100 x 3 mm) using MS/MS detection (Atmospheric Pressure Ionization Tandem Mass Spectroscopy; Appendix 2, Figure 3). The mobile phase conditions for the separation consisted of 0.1% acetic acid in water:0.1% acetic acid in acetonitrile (90:10 to 10:90 to 90:10, v:v). The retention times were 7:35 minutes for NOA-447204 and NOA-407854, 7:98 minutes for CGA-153433, 8:40 minutes for pinoxaden, and 9:45 minutes for cloquintocet-mexyl.

The analytical purities of the reference standards were 96.4-99.6% for pinoxaden, 97.5-97.7% for NOA-407854, 94.5-97.8% for NOA-447204, 95.2-96.2% for cloquintocet-mexyl, and 99.2-99.8% for CGA-153433 (Appendix 2, Figure 1).

A method validation study was conducted prior to the analysis of the test samples using 0-15 cm depth control soil samples that was fortified in triplicate with pinoxaden, NOA-407854, and NOA-447204 at concentrations of 0.50, 5.0, and 50 ppb. Mean recoveries of pinoxaden were 70.7%, 84.3%, and 96.7% for the 0.50, 5.0, and 50 ppb fortification levels, respectively;

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corresponding mean recoveries of NOA-407854 and NOA-447204 were 75.2-79.1%, 98.0-102%, and 118-120% for the three fortification levels (Appendix 2, Table 1).

## II. RESULTS AND DISCUSSION

**1. APPLICATION MONITORS:** The study author stated that the laboratory could not achieve acceptable recoveries for fortified procedural recovery samples analyzed concurrently with the tank mix samples, and that the tank mix samples and application monitors were not analyzed.

**2. RECOVERY FROM FIELD SPIKES:** The study author stated that the field spike samples were not analyzed as a result of a change in personnel at the analytical laboratory.

**3. MASS ACCOUNTING:** A mass balance was not determined.



Table 7. Mean concentration of pinoxaden and cloquintocet-mexyl residues expressed as ppb soil in the bare ground plot (n=3).

Compound	Soil depth (cm)	Sampling times (days)														
		0	0.5	1	3	7	14	21	31	38	60	91	124	185	274	389
Pinoxaden (NOA-407855)	0-15	5.9	5.1	2.5	2.5	0.87	0.67	0.61	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	15-30	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	30-45	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
NOA-407854	0-15	14	14	14	11	8.5	5.7	4.0	1.3*	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	15-30	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	30-45	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
NOA-447204	0-15	0.55*	0.54*	1.0	1.2	1.1	0.96	2.3	1.3	1.0	0.98	1.0	0.62*	<0.50	<0.50	<0.50
	15-30	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	1.4	0.89*	0.75	0.84*	0.97	<0.50	<0.50	<0.50
	30-45	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	0.58*	<0.50	<0.50	0.52*	0.57*	<0.50	<0.50	<0.50
Cloquintocet-mexyl (CGA-185072)	0-15	4.2	4.2	2.9	2.4	2.3	1.5	2.7	0.51*	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	15-30	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	30-45	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
CGA-153433	0-15	<0.50	0.51*	0.69*	0.79	0.54*	0.77	1.1	1.6	0.60	0.61*	<0.50	<0.50	<0.50	<0.50	<0.50
	15-30	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	30-45	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50

Data were obtained from Tables 2a-2c, pp. 34-36 of the study report. Mean values are averages of three replicates. The method LOQ was 0.5 ppb for each analyte in soil. Total extractable and non-extractable residues and total recovery were not determined.

\* Residue values above the limit of quantitation were observed in only one or two of the three replicate samples. The average value was calculated by substituting 0.5 ppb for the replicate residue levels that were <0.5 ppb.

NS = no sample.

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Table 8. Mean concentration of pinoxaden and cloquintocet-mexyl residues expressed as ppb soil in the cropped plot (n=3).

Compound	Soil depth (cm)	Sampling times (days)														
		0	0.5	1	3	7	14	21	31	38	60	91	124	185	274	389
Pinoxaden (NOA-407855)	0-15	7.6	7.1	3.7	2.3	2.9	0.71	0.80	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	15-30	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	30-45	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
NOA-407854	0-15	12	13	12	13	6.6	3.6	1.7	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	15-30	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	30-45	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
NOA-447204	0-15	<0.50	0.53*	1.2	1.7	1.7	1.4	1.1	1.2	0.58*	0.59*	<0.50	0.51*	0.50*	<0.50	<0.50
	15-30	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	1.1	0.83	0.66*	0.59	0.51*	0.50*	<0.50	<0.50
	30-45	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	0.79 <sup>1</sup>	0.51*	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
Cloquintocet-mexyl (CGA-185072)	0-15	3.9	3.9	2.1	2.2	2.6	2.9	2.5	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	15-30	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	30-45	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
CGA-153433	0-15	0.52*	0.56*	0.70	0.86	0.77	1.1	0.91	0.51*	<0.50	0.50*	0.68	0.55*	<0.50	<0.50	<0.50
	15-30	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	30-45	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50

Data were obtained from Tables 1a-1c, pp. 31-33 of the study report. Mean values are averages of three replicates. The method LOQ was 0.5 ppb for each analyte in soil. Total extractable and non-extractable residues and total recovery were not determined.

\* Residue values above the limit of quantitation were observed in only one or two of the three replicate samples. The average value was calculated by substituting 0.5 ppb for the replicate residue levels that were <0.5 ppb.

<sup>1</sup> Replicate B only, data from replicates A and C were not reportable.

NS = no sample.

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**4. PARENT COMPOUND:** In the bare ground plot, the measured zero-time concentration in the 0-15 cm soil depth was 5.9 ppb, which is 15% of the applied rate (reviewer-calculated based on a theoretical day-0 concentration of 0.039 mg a.i./kg in the 0-15 cm soil depth; Table 2a). The day-0 concentration of pinoxaden + NOA-407854 (after converting to parent equivalents) was 61% of the applied rate (reviewer-calculated). Following day 0, pinoxaden decreased to 2.5 ppb by 1 day and was last detected above the LOQ at 0.61 ppb at 21 days posttreatment. Pinoxaden was not detected above the LOQ in soil below the 0-15 cm depth (Tables 2b-2c). The reviewer-calculated half-life of pinoxaden in soil under terrestrial field conditions was 7 days ( $r^2 = 0.73$ ), based on all data points for the top 0-15 cm soil layer. The half-life value was calculated using linear regression analysis performed on a plot of ln-transformed concentrations vs. time and the equation  $t_{1/2} = \ln 2 / k$ , where  $k$  is the rate constant. A DT90 value was not calculated. However, the reviewer notes that the dissipation pattern of pinoxaden was bi-phasic, and that the observed half-life was approximately 1 day.

In the cropped plot, the maximum measured concentration in the 0-15 cm soil depth was 7.6 ppb at day 0, which is 19% of the applied rate (reviewer-calculated based on a theoretical day-0 concentration of 0.040 mg a.i./kg in the 0-15 cm soil depth; Table 1a). The maximum concentration of pinoxaden + NOA-407854 (after converting to parent equivalents) was 59% of the applied rate at 0.5 days posttreatment (reviewer-calculated). Following day 0, pinoxaden decreased to 3.7 ppb by 1 day and was last detected above the LOQ at 0.80 ppb at 21 days posttreatment. Pinoxaden was not detected below the 0-15 cm soil depth (Tables 1b-1c). The reviewer-calculated half-life of pinoxaden in soil under terrestrial field conditions was 6 days ( $r^2 = 0.80$ ), based on all data points for the top 0-15 cm soil layer. The half-life value was calculated using linear regression analysis performed on a plot of ln-transformed concentrations vs. time and the equation  $t_{1/2} = \ln 2 / k$ , where  $k$  is the rate constant. A DT90 value was not calculated. However, the reviewer notes that the dissipation pattern of pinoxaden was bi-phasic and that the observed half-life was approximately 1 day.

**5. TRANSFORMATION PRODUCTS:** After converting transformation product values to parent equivalents by dividing by the corresponding molecular weight conversion factors (0.79 for NOA-407854 and 0.82 for NOA-447204; see Reviewer Comment 1 for sample calculation), **NOA-407854** (7H-pyrazolo[1,2-d][1,4,5]oxadiazepine-7,9(8H)-dione, 8-(2,6-diethyl-4-methylphenyl)tetrahydro-) was the only transformation product detected in the soil at a concentration greater than 10% of the applied, based on the theoretical amount expected to be found in soil (Tables 1a-2c). The minor transformation product **NOA-447204** (7H-pyrazolo[1,2-d][1,4,5]oxadiazepine-7,9(8H)-dione, 8-(2,6-diethyl-4-methylphenyl)tetrahydro-8-hydroxy-) was not detected above 3% of the applied pinoxaden at any sampling interval.

In the bare ground plot, **NOA-407854** was detected in the 0-15 cm soil depth at a maximum concentration of 14 ppb from 0 to 1 day (which is 45.4% of the applied pinoxaden, after converting to parent equivalents; Table 2a), then decreased to 8.5 ppb by 7 days and was last detected above the LOQ at 1.3 ppb at 31 days posttreatment. NOA-407854 was not detected above the LOQ in soil below the 0-15 cm depth (Tables 2b-2c). The registrant-calculated half-life value for NOA-407854 was 10 days, calculated using non-linear regression (Figure 8). **NOA-447204** was detected in the 0-15 cm soil depth at a maximum concentration of 2.3 ppb at 21 days

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(which is 7.2% of the applied pinoxaden, after converting to parent equivalents), and was last detected at 0.62 ppb at 124 days posttreatment. NOA-447204 was detected in the 15-30 cm soil depth at 0.75-1.4 ppb from 31-124 days, and was detected in the 30-45 cm soil depth at 0.52-0.58 ppb at 31, 91, and 124 days posttreatment. Samples were not analyzed below the 30-45 cm soil layer.

In the cropped plot, **NOA-407854** was detected in the 0-15 cm soil depth at a maximum concentration of 13 ppb at 0.5 and 3 days (which is 41.1% of the applied pinoxaden, after converting to parent equivalents; Table 1a), then decreased to 6.6 ppb by 7 days, and was last detected above the LOQ at 1.7 ppb at 21 days posttreatment. NOA-407854 was not detected above the LOQ in soil below the 0-15 cm depth (Tables 1b-1c). The registrant-calculated half-life value for NOA-407854 was 8.1 days, calculated using non-linear regression (Figure 4).

**NOA-447204** was detected in the 0-15 cm soil depth at a maximum concentration of 1.7 ppb at 3 and 7 days (which is 5.1% of the applied pinoxaden, after converting to parent equivalents), and was last detected at 0.50 ppb at 185 days posttreatment. NOA-447204 was detected in the 15-30 cm soil depth at 0.50-1.1 ppb from 31-185 days, and was detected in the 30-45 cm soil depth at 0.51-0.79 ppb at 31 and 38 days posttreatment. Samples were not analyzed below the 30-45 cm soil layer.

Table 9: Chemical names and CAS numbers for the transformation products of pinoxaden.

Applicant's Code Name	CAS Number	CAS Chemical Name	Chemical formula	Molecular weight	SMILES string
NOA-407854	314020-44-5	7H-Pyrazolo[1,2-d][1,4,5]oxadiazepine-7,9(8H)-dione, 8-(2,6-diethyl-4-methylphenyl)tetrahydro-	C <sub>18</sub> H <sub>24</sub> N <sub>2</sub> O <sub>3</sub>	316.5	Not available
NOA-447204	Not available	7H-Pyrazolo[1,2-d][1,4,5]oxadiazepine-7,9(8H)-dione, 8-(2,6-diethyl-4-methylphenyl)tetrahydro-8-hydroxy-	C <sub>18</sub> H <sub>24</sub> N <sub>2</sub> O <sub>4</sub>	328.5	Not available

Data were obtained from p. 24 and Appendix 2, Figure 1, pp. 270-271 of the study report. The chemical formulas and molecular weight of NOA-447204 were determined by the reviewer based on the structures presented in Appendix 2.

**6. EXTRACTABLE AND NON-EXTRACTABLE RESIDUES:** Non-extractable residues were not measured.

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Table 10: Dissipation routes of pinoxaden under field conditions.

Route of dissipation		% of applied amount (at the end of study period)	
		Bare ground plot	Cropped plot
Accumulation (residues) in soil/ carry over		0%	0%
Transformation (% of transformation products)		0%	0%
Leaching, if measured		Maximum depth detected	Maximum depth detected
	Pinoxaden	0-15 cm	0-15 cm
	NOA-407854	0-15 cm	0-15 cm
	NOA-447204	30-45 cm	30-45 cm
Volatilization, if measured		Not measured	Not measured
Plant uptake, if measured		Not applicable	Not measured
Run off, if measured		Not measured	Not measured
Total			

Data were obtained from Tables 1a-2c, pp. 31-36 the study report.

**7. VOLATILIZATION:** The concentration of applied pinoxaden lost through volatilization was not determined.

**8. PLANT UPTAKE:** Plant uptake was not determined.

**9. LEACHING:** Pinoxaden and NOA-407854 were not detected below the 0-15 cm soil depth in either test plot; however, NOA-447204 was detected in both test plots in all depths analyzed (0-15, 15-30, and 30-45 cm; Tables 1a-2c).

**10. RUN OFF:** Run off was not studied.

**11. RESIDUE CARRYOVER:** DT90 values were not calculated. By the last sampling interval, 389 days posttreatment, 0% of the applied parent compound was detected in the bare ground and cropped test plots and has no potential to carryover (Tables 1a-2c). No transformation products were detected above the LOQ at the end of the study period.

**12. SUPPLEMENTARY STUDY RESULTS:** Pinoxaden was not stable in frozen soil, with a mean corrected recovery of 101% at day 0 decreasing to 80% by 9 months and 69% by 12 months (MRID 46203022); recoveries were corrected for the mean procedural recovery. NOA-407854 and NOA-447204 were both stable over time, with mean corrected recoveries ranging from 95-108% and 98-116%, respectively, with no pattern of decline.

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**III. STUDY DEFICIENCIES:** Pinoxaden was not stable in frozen storage, decreasing to a corrected recovery of 69% following 12 months (from 101% at day 0; MRID 46203022). It is necessary to demonstrate the stability of the parent compound under typical storage conditions to ensure that the degradation of the parent occurred in the field and not during storage of the test samples. If pinoxaden was degrading during storage of the samples, then the calculated half-life value will not correctly reflect dissipation in the field.

### IV. REVIEWER'S COMMENTS:

1. To determine percentage of the applied values for the transformation products, transformation product concentration values were converted to parent equivalents by dividing by the corresponding molecular weight conversion factors (0.79 for NOA-407854 and 0.82 for NOA-447204). Molecular weight conversion factors are calculated by dividing the molecular weight of the transformation product by the molecular weight of the parent. For example, for NOA-407854, 316.5 g/mol divided by 400.5 g/mol equals 0.79). To convert transformation product concentrations to parent equivalents, divide the transformation product concentration by the molecular weight conversion factor. For example, for NOA-407854, 14 ppb (detection at day-0, bare ground plot, 0-15 cm soil depth) divided by 0.79 equals 17.7 ppb parent equivalents.

The percent of each transformation product in terms of percent of the applied pinoxaden was calculated by dividing the concentration of the transformation product in parent equivalents (see above on how to convert to parent equivalents) by the theoretical day-0 concentration of pinoxaden in the 0-15 cm soil depth (0.039 mg a.i./kg for the bare ground plot and 0.040 mg a.i./kg for the cropped plot; registrant-calculated; see footnote 1 to DER Table 5). For example, for NOA-407854, 17.7 ppb parent equivalents (detection at day-0, bare ground plot, 0-15 cm soil depth, see above for calculation) divided by 39  $\mu\text{g a.i./kg}$  = 0.454 or 45.4% of the applied pinoxaden.

2. The registrant-calculated half-life of pinoxaden in the bare ground and cropped plots was 2.1 days ( $r^2 = 0.87$ ) and 2.8 days ( $r^2 = 0.81$ ), respectively, based on non-linear first-order analysis (SigmaPlot™ 8.0 for Windows) and determined using residues from the 0-15 cm soil depth (Figures 3-3a; and Figures 7-7a).
3. The actual application rate could not be determined because the application monitors used to verify the application rate for both test plots were not analyzed; however, the reviewer notes that the day-0 concentration of pinoxaden + NOA-407854 (after converting to parent equivalents) was 61% of the applied rate for the bare ground plot and 59% of the applied for the cropped plot. Values are reviewer-calculated based on a theoretical day-0 concentration of 0.039 mg a.i./kg in the 0-15 cm soil depth for the bare ground plot and 0.040 mg a.i./kg for the cropped plot.

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4. The maximum depth of leaching could not be determined for the transformation product NOA-447204, which was detected in both the bare ground and cropped test plots at concentrations ranging from 0.51-0.79 ppb in the lowest soil depth analyzed (30-45 cm; Tables 1a-2c).
5. Mean procedural recoveries from soil samples that were fortified with pinoxaden, NOA-407854, and NOA-447204 at 0.5, 5.0, and 25 ppb and analyzed concurrently with samples from the bare ground plot were  $83.9 \pm 10.1\%$ ,  $101 \pm 11.5\%$ , and  $96.7 \pm 10.9\%$ , respectively (Appendix 2 and Tables 12-13). For the cropped plot, mean procedural recoveries were  $80.8 \pm 12.0\%$  for pinoxaden,  $105 \pm 13.0\%$  for NOA-407854, and  $93.6 \pm 13.9\%$  for NOA-447204 (Appendix 2, Tables 5-6).
6. Test samples from the treated bare ground and cropped plots were analyzed for residues of the safener, cloquintocet-mexyl and its transformation product CGA-153433 (acetic acid, [(5-chloro-8-quinolinyl)oxy]-; Appendix 2, Figure 1), and the results were included in DER Tables 7 and 8 by the reviewer. All other analytical results for the safener and its transformation product are reported below. Mean recoveries of cloquintocet-mexyl and CGA-153433 from method validation samples fortified with each analyte at 0.50, 5.0, and 50 ppb were  $99.5 \pm 7.6\%$  for cloquintocet-mexyl, and  $32.0 \pm 6.0\%$  for CGA-153433 (mean recoveries are across all three fortification levels; Appendix 2, Table 1). Mean procedural recoveries from soil samples fortified at 0.5, 5.0, and 25 ppb from the bare ground plot were  $81.1 \pm 7.8\%$  for cloquintocet-mexyl and  $34.3 \pm 19.9\%$  for CGA-153433, and mean recoveries from the cropped plot were  $83.9 \pm 11.0\%$  for cloquintocet-mexyl and  $33.0 \pm 24.6\%$  for CGA-153433 (Appendix 2; Tables 5-6; and Tables and 12-13). Recoveries of cloquintocet-mexyl and CGA-153433 from soil samples that were fortified at 5 ppb and stored frozen for up to 12 months ranged from 94-124% and 97-124%, respectively, indicating stability during storage (MRID 46203022).

The study author stated that, due to low recoveries (<40%) of CGA-153433 from the method validation samples and procedural recovery samples, the residue data reported for CGA-153433 should be considered qualitative only. The reviewer notes that the storage stability samples analyzed for CGA-153433 were analyzed according to a different analytical method.

### V. REFERENCES:

1. U.S. Environmental Protection Agency. 1982. Pesticide Assessment Guidelines, Subdivision N, Chemistry: Environmental Fate, Section 164-1, Terrestrial Field Dissipation Studies. Office of Pesticide and Toxic Substances, Washington, DC. EPA 540/9-82-021.
2. U.S. Environmental Protection Agency. 1993. Pesticide Registration Rejection Rate Analysis - Environmental Fate. Office of the Prevention, Pesticides, and Toxic Substances, Washington, DC. EPA 738-R-93-010.
3. U.S. Environmental Protection Agency. 1989. FIFRA Accelerated Reregistration, Phase 3

## **Data Evaluation Report on the terrestrial field dissipation of pinoxaden**

PMRA Submission Number {.....}

EPA MRID Number 46203025

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Technical Guidance. Office of the Prevention, Pesticides, and Toxic Substances,  
Washington, DC. EPA 540/09-90-078.



# **Data Evaluation Report on the terrestrial field dissipation of pinoxaden**

PMRA Submission Number {.....}

EPA MRID Number 46203025

## **Attachment 1**

### **Structures of Parent and Transformation Products**

## Data Evaluation Report on the terrestrial field dissipation of pinoxaden

PMRA Submission Number {.....}

EPA MRID Number 46203025

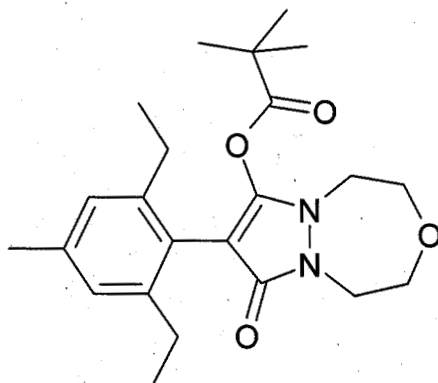
### Pinoxaden [NOA-407855]

**IUPAC name:** 8-(2,6-Diethyl-p-tolyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl 2,2-dimethylpropanoate.

**CAS name:** 8-(2,6-Diethyl-4-methylphenyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl 2,2-dimethylpropanoate.  
8-(2,6-Diethyl-4-methylphenyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl ester 2,2-dimethylpropanoic acid.

**CAS No:** 243973-20-8.

**SMILES string:** O=C1C(=C(N2N1CCOCC2)OC(=O)C(C)(C)C)c1c(cc(cc1CC)C)CC.



# **Data Evaluation Report on the terrestrial field dissipation of pinoxaden**

PMRA Submission Number {.....}

EPA MRID Number 46203025

## **Identified Compounds**

## Data Evaluation Report on the terrestrial field dissipation of pinoxaden

PMRA Submission Number {.....}

EPA MRID Number 46203025

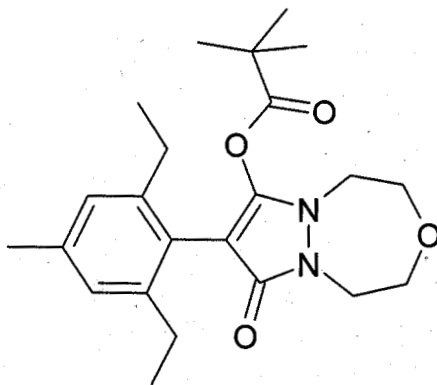
### Pinoxaden [NOA-407855]

**IUPAC name:** 8-(2,6-Diethyl-p-tolyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl 2,2-dimethylpropionate.

**CAS name:** 8-(2,6-Diethyl-4-methylphenyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl 2,2-dimethylpropanoate.  
8-(2,6-Diethyl-4-methylphenyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl ester 2,2-dimethylpropanoic acid.

**CAS No:** 243973-20-8.

**SMILES string:** O=C1C(=C(N2N1CCOCC2)OC(=O)C(C)(C)C)c1c(cc(cc1CC)C)CC.



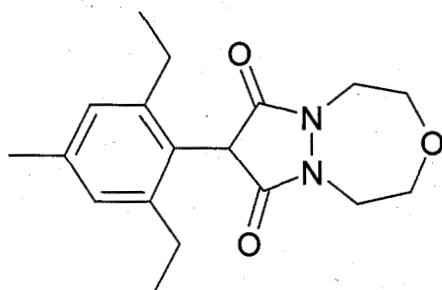
# Data Evaluation Report on the terrestrial field dissipation of pinoxaden

PMRA Submission Number {.....}

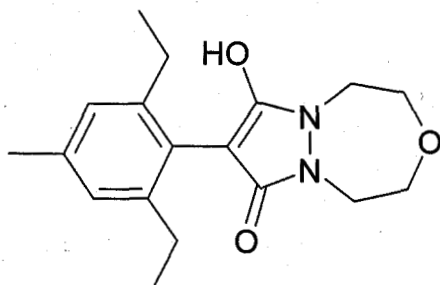
EPA MRID Number 46203025

NOA-407854

**IUPAC name:** 8-(2,6-Diethyl-4-methyl-phenyl)tetrahydro-pyrazolo[1,2-d][1,4,5]oxadiazepin-7,9-dione.  
**CAS name:** 8-(2,6-Diethyl-4-methylphenyl)tetrahydro-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-7,9(8H)-dione.  
8-(2,6-Diethyl-4-methylphenyl)-1,2,4,5-tetrahydro-9-hydroxy-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-7-one.  
**CAS No:** 314020-44-5 (keto form); 243973-19-5 (enol form).



Keto form



Enol form

## Data Evaluation Report on the terrestrial field dissipation of pinoxaden

PMRA Submission Number {.....}

EPA MRID Number 46203025

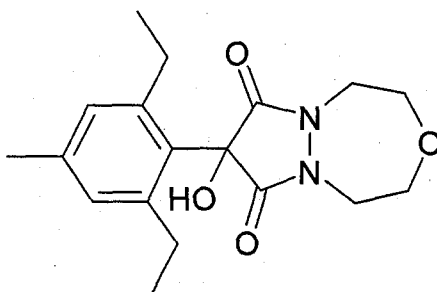
**NOA-447204**

**IUPAC name:** 8-(2,6-Diethyl-4-methyl-phenyl)-8-hydroxy-tetrahydro-pyrazolo[1,2-d][1,4,5]oxadiazepin-7,9-dione.

**CAS name:** 8-(2,6-Diethyl-4-methylphenyl)tetrahydro-8-hydroxy-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-7,9(8H)-dione.

**CAS No:** Not reported.

**SMILES string:** O=C1C(c2c(cc(cc2CC)C)CC)(C(=O)N2N1CCOCC2)O.



## **Data Evaluation Report on the terrestrial field dissipation of pinoxaden**

**PMRA Submission Number {.....}**

**EPA MRID Number 46203025**

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**Structure of the Safener and its Transformation Product**

## Data Evaluation Report on the terrestrial field dissipation of pinoxaden

PMRA Submission Number {.....}

EPA MRID Number 46203025

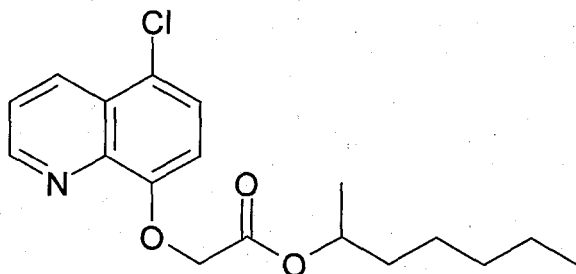
### CGA-185072 [Safener; Cloquintocet-mexyl]

**IUPAC name:** 5-Chloro-8-quinolinoxyacetic acid-1-methylhexylester. (Previously known in structure files; see Safener of Clodinfop-propargyl)

**CAS name:** 1-Methylhexyl ester [(5-chloro-8-quinolinyl)oxy]-acetic acid.

**CAS No:** 99607-70-2.

**SMILES string:** Clc1ccc(c2c1cccn2)OCC(=O)OC(CCCCC)C.





# **Data Evaluation Report on the terrestrial field dissipation of pinoxaden**

PMRA Submission Number {.....}

EPA MRID Number 46203025

## **Identified Compounds**

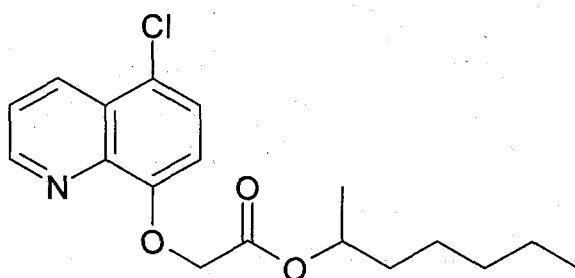
## Data Evaluation Report on the terrestrial field dissipation of pinoxaden

PMRA Submission Number {.....}

EPA MRID Number 46203025

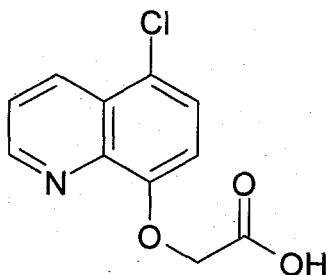
### CGA-185072 [Safener; Cloquintocet-mexyl]

**IUPAC name:** 5-Chloro-8-quinolinoxyacetic acid-1-methylhexylester. (Previously known in structure files; see Safener of Clodinfop-propargyl)  
**CAS name:** 1-Methylhexyl ester [(5-chloro-8-quinolinyl)oxy]-acetic acid.  
**CAS No:** 99607-70-2.  
**SMILES string:** Clc1ccc(c2c1cccn2)OCC(=O)OC(CCCCC)C.



### CGA 153433

**IUPAC name:** Not reported.  
**CAS name:** [(5-Chloro-8-quinolinyl)oxy]-acetic acid.  
**CAS No:** 88349-88-6.  
**SMILES string:** Clc1ccc(c2c1cccn2)OCC(=O)O.



# **Data Evaluation Report on the terrestrial field dissipation of pinoxaden**

PMRA Submission Number {.....}

EPA MRID Number 46203025

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## **Attachment 1**

### **Structures of Parent and Transformation Products**

## Data Evaluation Report on the terrestrial field dissipation of pinoxaden

PMRA Submission Number {.....}

EPA MRID Number 46203025

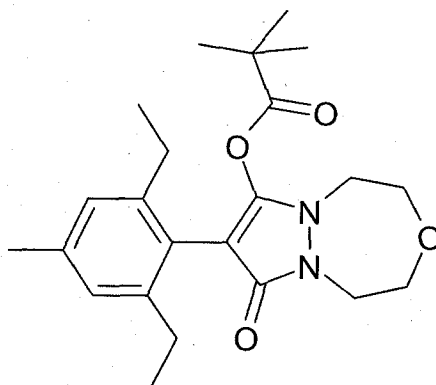
### Pinoxaden [NOA-407855]

**IUPAC name:** 8-(2,6-Diethyl-p-tolyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl 2,2-dimethylpropionate.

**CAS name:** 8-(2,6-Diethyl-4-methylphenyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl 2,2-dimethylpropanoate.  
8-(2,6-Diethyl-4-methylphenyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl ester 2,2-dimethylpropanoic acid.

**CAS No:** 243973-20-8.

**SMILES string:** O=C1C(=C(N2N1CCOCC2)OC(=O)C(C)(C)C)c1c(cc(cc1CC)C)CC.



# **Data Evaluation Report on the terrestrial field dissipation of pinoxaden**

PMRA Submission Number {.....}

EPA MRID Number 46203025

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## **Identified Compounds**

## Data Evaluation Report on the terrestrial field dissipation of pinoxaden

PMRA Submission Number {.....}

EPA MRID Number 46203025

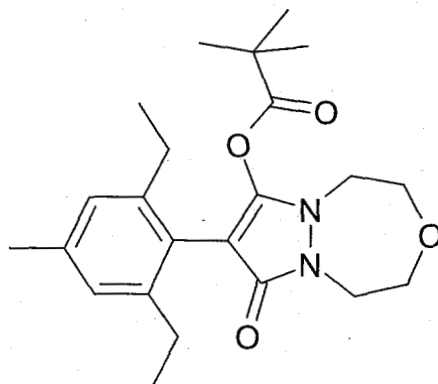
### Pinoxaden [NOA-407855]

**IUPAC name:** 8-(2,6-Diethyl-p-tolyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl 2,2-dimethylpropionate.

**CAS name:** 8-(2,6-Diethyl-4-methylphenyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl 2,2-dimethylpropanoate.  
8-(2,6-Diethyl-4-methylphenyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl ester 2,2-dimethylpropanoic acid.

**CAS No:** 243973-20-8.

**SMILES string:** O=C1C(=C(N2N1CCOCC2)OC(=O)C(C)(C)C)c1c(cc(cc1CC)C)CC.



## Data Evaluation Report on the terrestrial field dissipation of pinoxaden

PMRA Submission Number {.....}

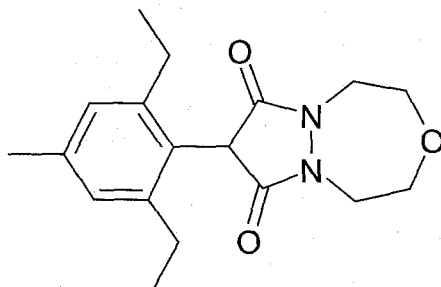
EPA MRID Number 46203025

**NOA-407854**

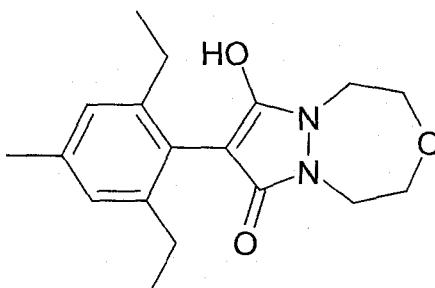
**IUPAC name:** 8-(2,6-Diethyl-4-methyl-phenyl)tetrahydro-pyrazolo[1,2-d][1,4,5]oxadiazepin-7,9-dione.

**CAS name:** 8-(2,6-Diethyl-4-methylphenyl)tetrahydro-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-7,9(8H)-dione.  
8-(2,6-Diethyl-4-methylphenyl)-1,2,4,5-tetrahydro-9-hydroxy-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-7-one.

**CAS No:** 314020-44-5 (keto form); 243973-19-5 (enol form).



Keto form



Enol form

## Data Evaluation Report on the terrestrial field dissipation of pinoxaden

PMRA Submission Number {.....}

EPA MRID Number 46203025

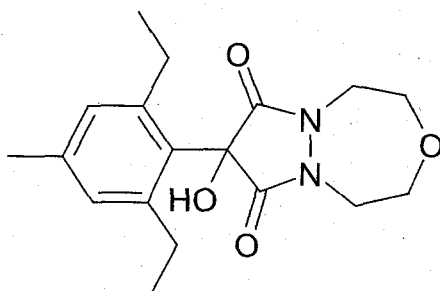
**NOA-447204**

**IUPAC name:** 8-(2,6-Diethyl-4-methyl-phenyl)-8-hydroxy-tetrahydro-pyrazolo[1,2-d][1,4,5]oxadiazepin-7,9-dione.

**CAS name:** 8-(2,6-Diethyl-4-methylphenyl)tetrahydro-8-hydroxy-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-7,9(8H)-dione.

**CAS No:** Not reported.

**SMILES string:** O=C1C(c2c(cc(cc2CC)C)CC)(C(=O)N2N1CCOCC2)O.





## **Data Evaluation Report on the terrestrial field dissipation of pinoxaden**

PMRA Submission Number {.....}

EPA MRID Number 46203025

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Structure of the Safener and its Transformation Product

## Data Evaluation Report on the terrestrial field dissipation of pinoxaden

PMRA Submission Number {.....}

EPA MRID Number 46203025

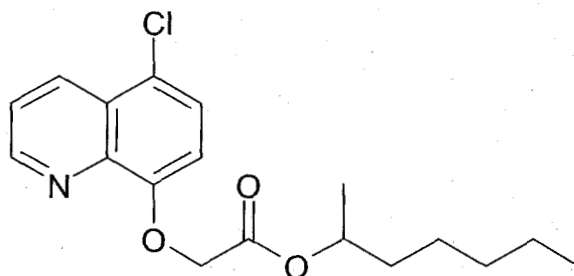
### CGA-185072 [Safener; Cloquintocet-mexyl]

**IUPAC name:** 5-Chloro-8-quinolinoxyacetic acid-1-methylhexylester. (Previously known in structure files; see Safener of Clodinfop-propargyl)

**CAS name:** 1-Methylhexyl ester [(5-chloro-8-quinolinyl)oxy]-acetic acid.

**CAS No:** 99607-70-2.

**SMILES string:** Clc1ccc(c2c1cccn2)OCC(=O)OC(CCCCC)C.



# **Data Evaluation Report on the terrestrial field dissipation of pinoxaden**

PMRA Submission Number {.....}

EPA MRID Number 46203025

---

## **Identified Compounds**

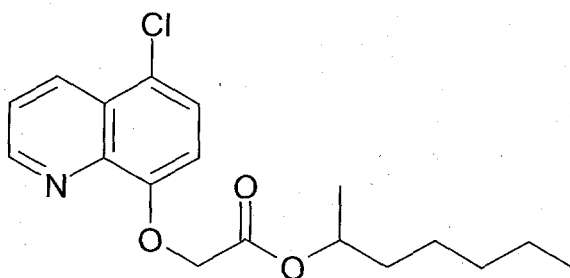
## Data Evaluation Report on the terrestrial field dissipation of pinoxaden

PMRA Submission Number {.....}

EPA MRID Number 46203025

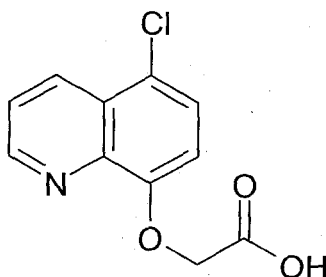
### CGA-185072 [Safener; Cloquintocet-mexyl]

**IUPAC name:** 5-Chloro-8-quinolinoxyacetic acid-1-methylhexylester. (Previously known in structure files; see Safener of Clodinfop-propargyl)  
**CAS name:** 1-Methylhexyl ester [(5-chloro-8-quinolinyl)oxy]-acetic acid.  
**CAS No:** 99607-70-2.  
**SMILES string:** Clc1ccc(c2c1cccn2)OCC(=O)OC(CCCCC)C.



### CGA 153433

**IUPAC name:** Not reported.  
**CAS name:** [(5-Chloro-8-quinolinyl)oxy]-acetic acid.  
**CAS No:** 88349-88-6.  
**SMILES string:** Clc1ccc(c2c1cccn2)OCC(=O)O.



Attachment 2  
Excel Spreadsheets

Chemical Name Pinoxaden  
 PC Code 147500  
 MRID 46203025  
 Guideline No. 164-1

**Bare-ground plot**

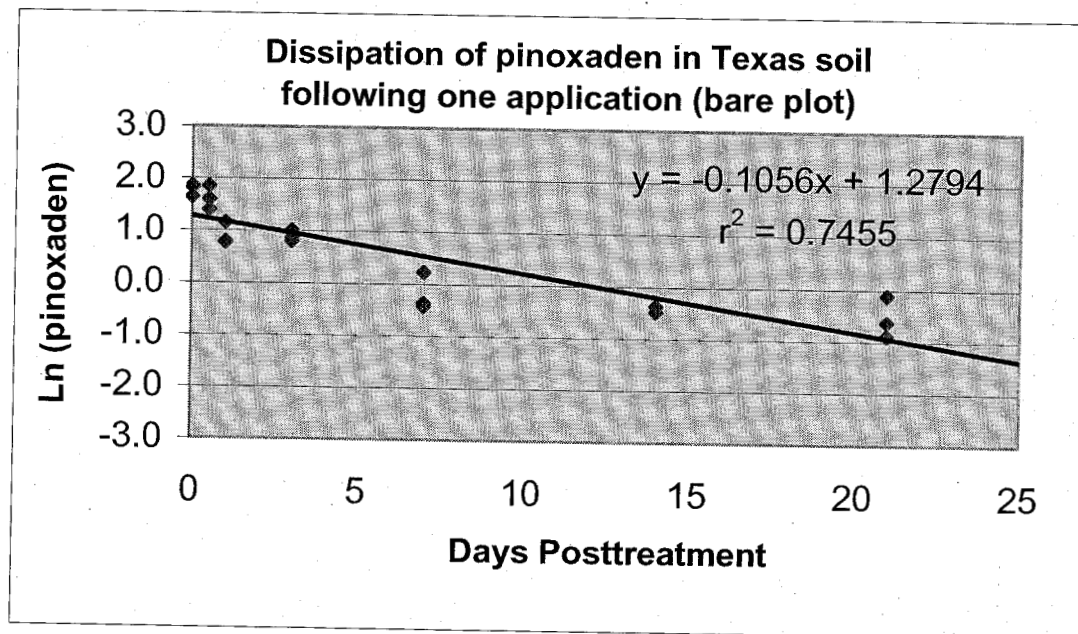
Half-life (days) = 7

Half-life calculated using all replicate data

Days Posttreatment	Pinoxaden (ppb)	Ln (pinoxaden)
0	5.14	1.6371
0	6.40	1.8563
0	6.18	1.8213
0.5	4.02	1.3913
0.5	6.35	1.8485
0.5	4.93	1.5953
1	3.17	1.1537
1	2.16	0.7701
1	2.20	0.7885
3	2.39	0.8713
3	2.76	1.0152
3	2.23	0.8020
7	0.694	-0.3653
7	1.26	0.2311
7	0.653	-0.4262
14	0.693	-0.3667
14	0.687	-0.3754
14	0.618	-0.4813
21	0.895	-0.1109
21	0.410	-0.8916
21	0.534	-0.6274
31	<0.50	
31	<0.50	
31	<0.50	

Data obtained from Appendix 2, Table 9, pp. 227-236 in the study report.

\* Replicate values are reviewer-calculated means of multiple analyses where replicates were re-analyzed.



Chemical Name Pinoxaden  
 PC Code 147500  
 MRID 46203025  
 Guideline No. 164-1

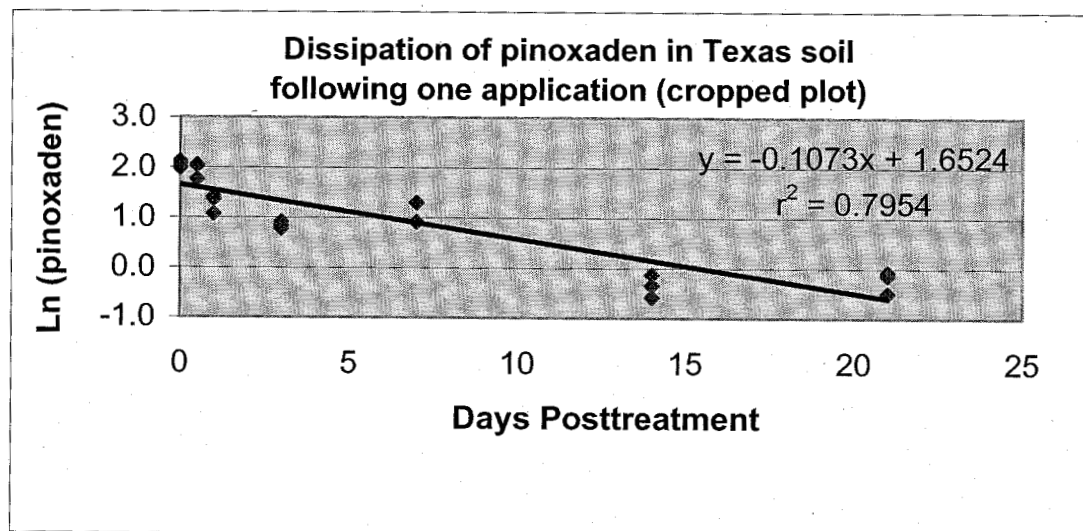
Cropped plot

Half-life (days) = 6  
 Half-life calculated using all replicate data

Days Posttreatment	Pinoxaden (ppb)	Ln (pinoxaden)
0	7.92	2.0694
0	7.29	1.9865
0	8.33	2.1199
0.5	5.82	1.7613
0.5	7.61	2.0295
0.5	7.71	2.0425
1	3.92	1.3661
1	4.10	1.4110
1	2.92	1.0716
3	2.17	0.7747
3	2.48	0.9083
3	2.31	0.8372
7	2.50	0.9163
7	3.67	1.3002
7	2.49	0.9123
14	0.875	-0.1335
14	0.708	-0.3453
14	0.557	-0.5852
21	0.607	-0.4992
21	0.867	-0.1427
21	0.930	-0.0726
31	<0.50	
31	<0.50	
31	<0.50	

Data obtained from Appendix 2, Table 2, pp. 193-201 in the study report.

\* Replicate values are reviewer-calculated means of multiple analyses where replicates were re-analyzed.



**Pages 48-64 \*Access to FIFRA health and safety data is restricted under FIFRA section 10(g)\***